



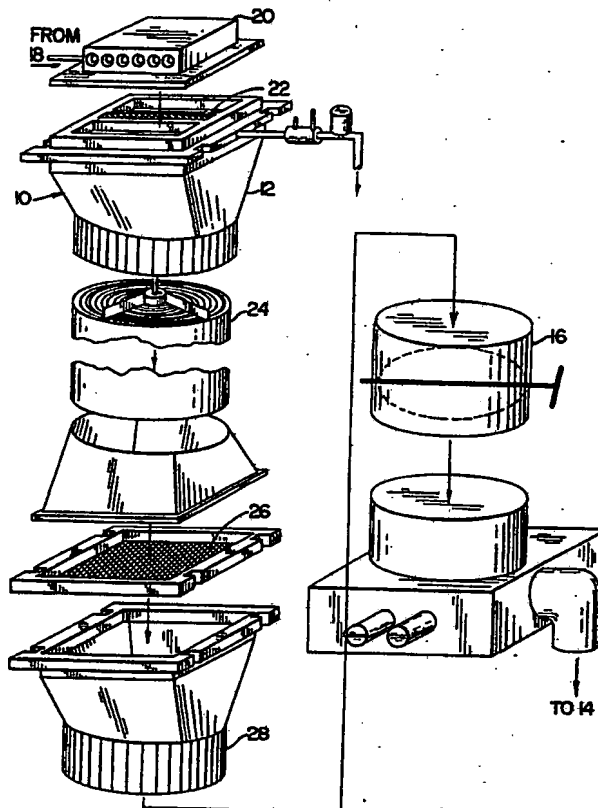
## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification <sup>5</sup> : <b>B01D 35/157, 51/10</b>	<b>A1</b>	(11) International Publication Number: <b>WO 93/06910</b>
		(43) International Publication Date: <b>15 April 1993 (15.04.93)</b>
<p>(21) International Application Number: <b>PCT/US92/08532</b></p> <p>(22) International Filing Date: <b>7 October 1992 (07.10.92)</b></p> <p>(30) Priority data: <b>772,905</b>      <b>8 October 1991 (08.10.91)</b>      <b>US</b></p> <p>(71) Applicant: <b>UNITED STATES ENVIRONMENTAL PROTECTION AGENCY [US/US]; 401 M Street, S.W., Washington, DC 20460 (US).</b></p> <p>(72) Inventor: <b>BURTON, Robert, M. ; 2108 Kipawa Street, Raleigh, NC 27607 (US).</b></p> <p>(74) Agents: <b>HEARD, Joseph, H. et al.; Bell, Seltzer, Park &amp; Gibson, P.O. Drawer 34009, Charlotte, NC 28234 (US).</b></p>		<p>(81) Designated States: <b>AT, AU, BB, BG, BR, CA, CH, CS, DE, DK, ES, FI, GB, HU, JP, KP, KR, LK, LU, MG, MN, MW, NL, NO, PL, RO, RU, SD, SE, UA, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, SN, TD, TG).</b></p> <p><b>Published</b> <i>With international search report.</i></p>

(54) Title: METHOD AND APPARATUS FOR SAMPLING AIR CONTAMINANTS

## (57) Abstract

A sampling apparatus (10) for separating and collecting contaminants contained in an air sample has a first particle separator (18), a second particle separator (20), a coarse filter (22), a denuder (24), a fine filter (26), a semi-volatile organic compound collector (28), a flow rate control device (16), and a vacuum pump (14). The apparatus (10) separates the coarse, intermediate, and fine particles, acid gases, basic gases, volatile organic compounds, and semi-volatile organic compounds at a high flow rate.



**FOR THE PURPOSES OF INFORMATION ONLY**

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AT	Austria	FR	France	MR	Mauritania
AU	Australia	GA	Gabon	MW	Malawi
BB	Barbados	GB	United Kingdom	NL	Netherlands
BE	Belgium	GN	Guinea	NO	Norway
BF	Burkina Faso	GR	Greece	NZ	New Zealand
BG	Bulgaria	HU	Hungary	PL	Poland
BJ	Benin	IE	Ireland	PT	Portugal
BR	Brazil	IT	Italy	RO	Romania
CA	Canada	JP	Japan	RU	Russian Federation
CF	Central African Republic	KP	Democratic People's Republic of Korea	SD	Sudan
CG	Congo	KR	Republic of Korea	SE	Sweden
CH	Switzerland	LI	Liechtenstein	SK	Slovak Republic
CI	Côte d'Ivoire	LK	Sri Lanka	SN	Senegal
CM	Cameroon	LU	Luxembourg	SU	Soviet Union
CS	Czechoslovakia	MC	Monaco	TD	Chad
CZ	Czech Republic	MG	Madagascar	TC	Togo
DE	Germany	ML	Mali	UA	Ukraine
DK	Denmark	MN	Mongolia	US	United States of America
ES	Spain			VN	Viet Nam
FI	Finland				

## METHOD AND APPARATUS FOR SAMPLING AIR CONTAMINANTS

### Field of the Invention

This invention relates generally to sampling air for contaminants, and more particularly relates to using an integrated sampler for identifying and measuring  
5 all contaminants in an atmospheric sample with a single device.

### Background of the Invention

The sampling of atmospheric contaminants is an ever-expanding area of research. Accurate  
10 identification, classification, and measurement of atmospheric contaminants is critical for, among other things, compliance with governmental regulations, studying the effect of contaminants on the surrounding environment, and assessment of air quality.

15 Sampling of air contaminants is a complex operation, as a given atmospheric sample may contain a multitude of contaminants that must be identified and measured. Problems have developed as researchers have attempted to sample large numbers of contaminants in a  
20 single operation; often the collection and/or measurement method for one component of a contaminated airstream adversely affects the measurement accuracy of other airstream components. Another difficulty is obtaining a sufficient sample of low concentration contaminants for  
25 proper analysis. High flow rate samplers reduce the time necessary to obtain an adequate contaminant sample, but

-2-

to date a completely integrated high flow rate sampler has not been available.

What is needed is a air sampler that is portable, able to accurately collect many contaminants simultaneously, do so quickly and easily, and provide samples that are easily measured.

#### Summary of the Invention

A first aspect of the invention is an apparatus for separating and collecting particle, volatile organic (VOCs), semi-volatile organic (SVOCs), acid gas, and basic gas components of an atmospheric sample directed along a path of travel in a downstream direction. The apparatus comprises a housing through which the sample travels, vacuum means for drawing the air sample through the housing, means for separating the particle components of the sample into a large particle part, an coarse part, and a fine part, filter means for trapping the intermediate and fine particle component parts, denuder means for removing gaseous contaminants, and collection means for collecting the semi-volatile organic components. Flow rate control means for controlling the flow rate of the airstream created by the vacuum means is also included. The apparatus can include a plurality of coarse filter means, denuders, fine filter means, and SVOC collection means through which branch streams of the sample travel. Preferably these branch flows are substantially equivalent in flow volume.

A second aspect of the invention is a method of sampling the contaminants in an air sample. The method comprises drawing the sample through a housing, separating the large particles of the sample, separating and trapping the coarse particles, removing the gaseous components in denuder means, trapping the fine particles, and removing the SVOCs. A preferred embodiment of the method includes dividing the sample into branch flows prior to trapping the coarse particles, removing and collecting the gaseous components, removing the fine

-3-

particles, and collecting SVOCs. The sample branch flows preferably are substantially equivalent in flow volume.

A third aspect of the invention is a method for determining the VOC concentration in an air sample. The method comprises the steps of dividing the sample into a plurality of branch flows, passing at least one branch flow through denuder means that removes VOCs, concurrently passing at least one branch flow through denuder means that does not remove VOCs, passing each branch flow through a separate particle filter means, passing each branch flow through a separate SVOC collection means, and removing and analyzing the composition and concentration of the filtrate on the fine filter means and the components trapped by the SVOC collection means for each branch flow.

#### Brief Description of the Drawings

Figure 1 is a flow chart indicating the path of an air sample through the sampler.

Figure 2 is a schematic representation of the sampler in which the sample remains in a single flow path.

Figure 3 is a schematic representation of the sampler in which the sample is divided into four branch flows at the denuders and in which the flow control means is an electronically controlled volumetric flow-based controller.

Figure 4 is a cross-sectional view of an SVOC polymer trap and its surrounding housing.

#### Detailed Description of the Invention

The sampling apparatus identified in the drawings by the numeral 10 separates and classifies particulate, gaseous organic (VOC), particulate organic, semi-volatile organic (SVOC), acid gas, and basic gas components of an ambient air sample at a high flow rate. Apparatus 10 is portable and thus can be transported easily to any location where testing is desired. The apparatus includes a housing 12, vacuum

-4-

means 14 that draws the air sample through the housing in a downstream direction, flow rate control means 16 for controlling the flow rate of the air sample, and a plurality of sampling devices. The sampling devices  
5 include a first particle separation means 18 for separating the particle components of the sample into a large particle part and a remaining part, a second particle separation means 20 for separating the remaining part of the particle component into an coarse  
10 part and a fine part, coarse filter means 22 for trapping the coarse part of the particle components, denuder means 24 for removing from the sample one or more sample components from the group of acid gas components, basic gas components, and volatile organic  
15 compound components (VOCs), fine filter means 26 for trapping the fine part of the particle component of the sample, and semi-volatile organic compound collection means 28 for collecting the semi-volatile organic compound (SVOCs) components from the air sample. The  
20 sample is drawn by the vacuum means sequentially through the large particle separation means, the coarse particle separation means, the coarse particle filter, the denuder means, the fine particle filter means, and the SVOC collection means before exiting the housing.

25 Vacuum means 14 draws air samples through housing 12 and each of the sampling devices located within. Vacuum means 14 is attached to housing 12 downstream of each of the sampling devices and may be of any pump-motor type capable of producing a  
30 sufficiently high flow rate to carry out the sampling operations.

The flow control means 16 is employed to ensure that the sample is passed through the sampler at a steady flow rate. The flow rate is critical because  
35 fluctuations in flow rate adversely affect the accuracy of the sampling results. Flow rate particularly affects the removal of gas components by the denuder

-5-

means 14, since only a specific flow rate will result in the proper face velocity along the denuder surfaces to allow the gas components to be removed.

Flow rate can be controlled manually or electronically. Manual control can be achieved by conventional control valves located downstream from the SVOC collection means and upstream from the vacuum means. Manual flow control means also requires a flow sensor within housing 12. Electronic control can be by an electronic flow controller such as that described in Behm et al., U.S. Patent No. 5,006,227. The electronic controller attaches to the motor of vacuum means 14 and adjusts the power output based on pressure readings taken inside the housing.

The first particle separation means 18 separates particles that are larger than would be suitable for any sampling within the sampler. Generally particles larger than 10  $\mu\text{m}$  are of no interest in a sampling because most of these particles are sufficiently large that they are screened out by the upper respiratory system and therefore cause no adverse health effects. The first particle separation means 18 is attached upstream of the other sampling components, and may be of any of various known types which can, without unduly impairing the flow rate of the sample, remove large particles from an airstream while allowing smaller particles to pass. It may employ commercially available filter media materials such as polytetrafluoroethylene (PTFE) coated glass fiber, quartz fiber, glass fiber, PTFE membrane, or Nucleopore membrane. Also suitable is any device configured to allow air passage but to restrict particle passage. An example of such a device is the two-stage inlet described in McFarland, Ortiz, and Bertich, A 10  $\mu\text{m}$  Cutpoint Size Selective Inlet for Hi-Vol Samplers, 34 J. Air Poll. Control Assoc., 544 (1984), which traps large particles in the convolutions

-6-

of its air passageway as the sample passes through.

The second particle separation means 20 divides the remaining sample contaminants into a coarse part and a fine part. It is generally necessary to  
5 remove the coarse particles from the air sample before passing the sample through denuder means 24, fine filter means 26, and SVOC collection means 28 for these downstream sampling elements to function properly. The second particle separation means 20 is attached to  
10 the housing 12 downstream from the first particle separation means 18.

Any type of separating device that will remove particles larger than the designated cut size, defined as the minimum-sized particle that will be  
15 separated from the sample, without unduly restricting flow rate, can be used. Exemplary coarse separation means include a high volume virtual impactor (HVVI) of the type described in Marple et al., U.S. Patent No. 4,670,135. The HVVI includes intake nozzles that  
20 extend perpendicular to the downstream flow direction created by the vacuum means. The size of the intake nozzles determines the sample cutpoint, which can be precisely predicted based on principles of fluid mechanics and particle dynamics. An exit tube for the  
25 major portion of the sample air flow extends perpendicular to the intake nozzles. Receiving tubes that accept the minor portion of the flow that carries particulate matter larger than the cutpoint extend parallel to and in line with the intake nozzles and  
30 lead into a chamber containing a particle filter.

The second separation means 20 may also include a plurality of separation means, each with a different cutpoint, to create different particle size samples within branch flows for different sampling  
35 treatment and particle characterization downstream. This can be accomplished by the use of a plurality of HHVIs. If multiple HHVIs are used, producing different



-7-

cutpoints within the same entering airstream requires that the intake nozzles for the separate HVVIs be sized according to the desired cutpoint.

The denuder means 24 is attached to the housing 12 downstream from the second separation means 20. It removes gaseous components from the sample. In general, the gas components of an air sample will include acid gasses, basic gasses and VOCs. Examples of acid gasses to be collected include  $\text{HNO}_2$ ,  $\text{HNO}_3$ ,  $\text{NO}_2$ ,  $\text{SO}_2$ , formic acid, and acetic acid. The basic gas of most interest is ammonia. VOCs to be collected include all other classes of gaseous organics, a large number of which are emitted from automotive exhaust, other combustion sources, and chemical processes. The denuder means can be configured to remove any or all of these compounds.

The denuder means 24 include a core material configured so that it presents large surface areas to the air sample traveling past it, and further includes a surface agent upon such surface areas of the core with an affinity for the class of sample to be removed. The core is shaped to expose all surface areas to the same face velocity as a sample is drawn over it; an exemplary configuration is concentric circles having their centers located in the center of a round housing and circumferences perpendicular to the path of sample travel. Examples of suitable core materials include aluminum and glass, but any material known in the sampling art is acceptable.

Suitable surface agents are known in the sampling art. If acid gasses are to be removed, a sodium carbonate film covering the surface areas of the denuder core is effective, although any base ordinarily employed in the sampling art may be used. If basic gasses are to be removed, an exemplary surface agent is citric acid, although any acid ordinarily employed in the sampling art may be used. If VOCs are to be

removed, an exemplary stripping material is silicon grease, although any hydrophobic compound ordinarily employed in the sampling art may be used. If the denuder means 24 is to remove two or three of these sample components, the denuders are arranged in series, with the acid gas denuders upstream of other denuders, the basic gas denuders downstream from any acid gas denuders, and organic gas denuders downstream of basic gas denuders. Removing acid gasses from the sample first prevents them from reacting with the VOCs during the sampling process.

It may be desirable to include a plurality of denuders in the samples. The denuding means 24 may be configured to accept branch flows created by a plurality of second particle separation means 20 or to create branch flows from a single main flow exiting the second particle separation means 20. In either case, each branch flow requires a separate denuder to receive and sample its contaminants exclusively; thus a plurality of denuders equal to the number of desired branch flows is required. It may be desired that these denuders each strip the same contaminants from the sample, in which case each contains the same denuding materials as the other denuders. In contrast, it may be desired that the individual denuders remove different contaminants, in which case the denuding material in one individual denuder will differ from that included in the others. Further, it may be desired not to remove certain contaminants from at least one sample branch flow, in which case the denuding structures for those contaminants are absent from the denuders through which those branches flow.

The fine filter means 26 is included in the sampler 10 to trap the fine particle part of the sample. The fine filter means 26 is attached to the housing 12 downstream from the denuder means 24. Any filter means currently employed in the sampling art

-9-

capable of trapping particles without unduly restricting the sample flow rate is suitable.

Exemplary filter media materials for the filter means include Nucleopore, quartz, PTFE, PTFE-coated glass  
5 fiber, or other membrane filter media; the choice of material may be influenced by the analytical procedure to be performed on the collected sample.

A plurality of filter sections may be employed; this is advantageous if different analytical  
10 tests are required to determine the composition and concentration of contaminants trapped by the filter. A plurality of filter sections also permits the use of different filter materials in each section if desired.

The SVOC collection means 28 are included in  
15 the sampler 10 to trap SVOCs and are attached to the housing 12 just downstream from the fine filter means 26. SVOCs attach to fine particles and thus are not stripped by the denuder means 24. Once the fine particles are trapped by the fine filter means 26, the  
20 continued air flow evaporates some of the attached SVOCs and causes them to pass through the fine filter means 26 and into the SVOC collection means. In general, SVOCs are organic compounds higher in molecular weight than VOCs; examples include  
25 polychlorinated biphenyl emissions (PCBs), pesticides, chlorinated dioxins, and nuclear aromatic hydrocarbons.

The SVOC collection means 28 includes a polymer trap for collecting the SVOCs. A polymer trap comprises a section of collecting material through  
30 which air can pass but which absorbs SVOCs. It can be made of a variety of materials known in the art to successfully trap SVOCs; examples of materials are polyurethane foam (PUFF), XAD-2, Tenax, and charcoal. It may be desirable to combine materials for heightened  
35 collection; an example is a polymer trap with a XAD-2 core sandwiched by outer layers of PUFF.

A plurality of SVOC collection means 28 may

-10-

also be employed. The benefit of multiple collection means is the ability to later perform different analytical procedures on each without affecting the results of another procedure. A plurality of filter  
5 media may also be used to facilitate sample analysis.

Operation of the sampler is represented by the flow chart of Figure 1. Power is supplied to the vacuum means 14, which creates a vacuum that draws an ambient air sample from the atmosphere into the housing  
10 12. The flow rate produced is monitored by the flow rate control means 16, which can be adjusted manually or electronically to maintain a steady sample flow rate. The large particle part of the particulate component of the sample is trapped by the first  
15 separation means 18. The remaining part of the sample then flows to the second particle separation means 20, which separates the coarse particles and directs them to the coarse filter means 22. The sample then flows into the denuder means 24. Precise flow rate control  
20 is critical in this removal step; the proper rate produces the required face velocity over the denuder surface and thus ensures removal of the desired gas component by the surface agent. If acid denuders are included, acid gasses are removed first, followed by  
25 removal of basic gasses if basic gas denuders are included, and further followed by VOC removal by VOC denuders if included in the sampler. The sample then flows through the fine filter means 26 where the fine particles are trapped, and then proceeds through the  
30 SVOC collection means 28, which removes SVOCs from the sample. Finally, the sample flows into and through the vacuum means 14 and exits into the surrounding atmosphere. Once sampling is complete, the sampling elements can be removed from the sampler 10 and the  
35 trapped components recovered from each element when possible for analysis of composition and concentration.

In a preferred embodiment of the sampler 30

-11-

shown in Figure 3, a 55 cfm pump 32 (Gast Manufacturing, Benton Harbor, MI 49022) (slightly modified to increase pump displacement and reduce friction losses) is connected to the housing 34 downstream of all sampling elements. The pump 32 is driven by a 3 Hp motor. An operating flow rate of at least 40 cfm within the housing 34 is preferred because it provides flow sufficient to rapidly collect a sufficiently large sample of each contaminant to effectively measure its concentration.

Pump operation is controlled electronically by the controller 40 described in Behm et al. This controller adjusts flow setting valves 42 attached downstream of each SVOC collector each 30 seconds based on local pressure sensed at the acceleration nozzle of an HVVI. The controller 40 also adjusts for barometric pressure and atmospheric temperature variations and thus reduces any error inflow rate that those parameters might cause.

The first particle separation means is a two-stage inlet 43 as described in McFarland, Ortiz, and Bertich. The inlet 43 is connected to the housing upstream of all of the sampling components. The cut point used for the large particle separation is 10  $\mu\text{m}$ ; particles larger than the cut point impinge in the convolutions of the inlet's flow tubes and must be cleared out periodically.

The second separation means comprises four HVVIs 44 connected to the housing downstream from the inlet. The cutpoints for the individual HVVIs are 0.32  $\mu\text{m}$ , 0.8  $\mu\text{m}$ , 1.5  $\mu\text{m}$ , and 2.5  $\mu\text{m}$ . Alternatively, all four have cutpoints of 2.5  $\mu\text{m}$ . The HVVIs are configured so that the flow volumes of the flows exiting the HVVIs are substantially equivalent.

The coarse filter means is a quartz fiber filter 46 located in the receiving chamber of the HVVI 44. Particles smaller than 10  $\mu\text{m}$  but larger than the

-12-

cutpoint of the HVVI 44 they entered are captured for analysis in this filter 46.

The denuder means is comprised of four denuders 50. Each of the denuders is aligned to receive a branch flow from one of the HVVIs 44. Acid gas denuders 52 comprise glass tubes dipped in a sodium carbonate solution. Basic gas denuders 54 comprise glass tubes dipped in a citric acid solution. VOC denuders 56 comprise a honeycombed configuration covered with silicon grease. After each sampling session, the denuder surface agents are removed and must be replaced. Three of the denuders remove acid gasses, basic gasses, and VOCs; the fourth denuder removes only acid and basic gasses. The exclusion of a VOC-removing structure from the fourth denuder allows phase distribution analysis to be performed on the VOCs to determine their concentration in the sample.

The fine filter means 60 is connected to the housing downstream from the denuder means 50 and contains four filter sections 62, one each of Nucleopore, PTFE, PTFE-coated glass fiber, and quartz. Each section 62 is aligned to receive one of the branch flows exiting the four denuders 50 described above and to trap its fine particles. These filter sections 62 can then be removed for analysis; each filter 62 is subjected to an appropriate analytical technique for that filter.

The SVOC collection means 70 is shown in Figure 4. SVOC collection means 70 is connected to the fine filter means 60 downstream from the fine filter means by a threaded pressure seal 72 and includes four polymer traps 74. The polymer traps include a XAD-core 76 sandwiched upstream and downstream by PUFF sections 78; this sandwich construction is surrounded by a glass cylinder 80, which in turn is surrounded by a steel housing 82. Each polymer trap 74 receives one branch flow exiting a section of the fine filter means 60.

-13-

The invention also comprises a method of separating and collecting contaminants in an air sample. The method comprises the steps of drawing an air sample through a housing, separating the particles  
5 in the sample into a large particle part and a remaining part, further separating particles in the remaining part into a coarse part and a fine part, removing from the sample at least one from the group comprising acid gas, basic gas, and VOC components,  
10 trapping the fine particles, and trapping the SVOCs.

The method can be used such that the contaminants of the sample remain in a single, main flow, or the contaminants can be divided into branch flows. The division can occur during the second  
15 separation step, when the coarse particles are separated from the fine particles, or during the gas component removal step. The employment of branch flows allows for the use of sampling procedures on one or more of the different branch flows that adversely  
20 affect procedures conducted on other contaminants.

If the sample is divided at the second separation step, utilization of a high volume virtual impactor can separate the fine particle components of the sample into branch flows based on the size of the  
25 particle. A preferred practice is the division of the fine particles into size ranges of 0-0.32  $\mu\text{m}$ , 0-0.8  $\mu\text{m}$ , 0-1.5  $\mu\text{m}$ , and 0-2.5  $\mu\text{m}$ . The sample is then divided into four branch flows, each carrying one of these fine particle size ranges.

30 The method also includes removal of some or substantially all of the gaseous components of the sample. Generally the utilization of any denuding means known in the sampling art to be specific for the gaseous component to be sampled is suitable. Any or  
35 all of the components comprising the group of acidic, basic, and organic gasses can be removed in this step. Utilization of sodium carbonate film on glass tubes for

-14-

removal of acid gasses is preferred; utilization of citric acid film on glass tubes for removal of basic acids is preferred; utilization of silicon grease on a honeycombed core structure for removal of organic  
5 gasses is preferred. In a preferred practice, all three denuding materials are included and aligned in series to remove substantially all of the gaseous components of the sample. Alternatively, it may be desirable to utilize a plurality of denuders to remove  
10 different components from different branch streams.

In removing the fine particles, almost any filter media that prevents particle passage while allowing air passage can be utilized. The method also may include the use of a plurality of filters, which  
15 permits the sampler to subject the filtrate captured on the separate filters to different analytical procedures.

Removal of the SVOCs of the sample requires passing the sample through a medium that can absorb  
20 these compounds without adversely affecting sample flow rate. Commonly used polymer traps include XAD-2, PUFF, charcoal, and Tenax. Like the fine particle removal step, it may be desirable to employ a plurality of SVOC collection devices to enable the sampler to perform  
25 different analytical procedures on the sample collected in each device.

A preferred practice of the method comprises passing the sample through a PM-10 two-step inlet to remove the large particles, passing the sample through  
30 four parallel HVVIs with cutpoints of 0.32  $\mu\text{m}$ , 0.8  $\mu\text{m}$ , 1.5  $\mu\text{m}$ , and 2.5  $\mu\text{m}$ , thus creating four branch streams. Coarse particles larger than the cutpoint are trapped in a glass fiber filter. Each branch stream is then passed through one of four parallel denuders containing  
35 glass tubes covered with sodium carbonate film to remove acid gasses, glass tubes covered with citric acid film to remove basic gasses, and a honeycombed



-15-

structure covered with silicon grease to remove VOCs. Each branch stream then passes through one of four fine filter sections: one section including quartz as the filter medium; one including teflon-coated glass fiber  
5 as the filter medium; one including Nucleopore as the filter medium, and one including PTFE as the filter medium. Each branch stream is finally passed through one of four polymer traps comprised of an XAD-2 core sandwiched by PUFF outer layers.

- 10 A third aspect of the invention is a method for determining the VOC concentration in an air sample. The method comprises the steps of dividing the sample into a plurality of branch flows, passing at least one branch flow through denuder means that removes VOCs,  
15 passing at least one branch flow through denuder means that does not remove VOCs, passing each branch flow through a separate particle filter means, passing each branch flow through a separate SVOC collection means, and removing and analyzing the composition and  
20 concentration of the filtrate on the fine filter means and the components trapped by the SVOC collection means for each branch flow. By comparing the amount of organic matter collected from a branch flow not  
25 subjected to a VOC denuder with a branch flow subjected to a VOC denuder, the VOC concentration of the sample can be determined.

-16-

## CLAIMS:

1. An apparatus for separating and collecting particle, gaseous organic, semi-volatile organic, acid gas, and basic gas components of an air sample directed along a path of travel in a downstream direction comprising:
- 5 (a) a housing;
- (b) vacuum means connected to said housing for drawing said air sample into and through said housing;
- 10 (c) first particle separation means for separating said particle components into a large particle part and a remaining part in an upstream section of said housing;
- (d) second particle separation means for
- 15 separating said remaining part of said particle component into a coarse part and a fine part, said second particle separation means being in a section of said housing downstream from said first particle separation means;
- 20 (e) coarse filter means for trapping said coarse part of said particle components, said coarse filter means being in a section of said housing downstream from said second particle separation means;
- (f) denuder means being in a section of said
- 25 housing downstream from said second particle separation means, said denuder means removing from the air sample one or more airstream components from the group consisting of said acid gas components, said basic gas components, and said volatile organic compound
- 30 components;
- (g) fine filter means for trapping said fine part of said particle component, said fine filter means being in a section of said housing downstream from said denuder means;
- 35 (h) SVOC collection means for collecting said SVOCs from said air sample, said SVOC collection

-17-

means being in a section of said housing downstream from said fine filter means;

(i) flow rate control means for controlling the flow rate of said air sample through said housing.

- 5                   2. An apparatus according to claim 1, in which said first particle separation means separates into said large particle part of said particle component all particles over 10  $\mu\text{m}$ .
- 10                   3. An apparatus according to claim 1, in which said first particle separation means is a two-step inlet.
4. An apparatus according to claim 3, in which said second particle separation means is a high volume virtual impactor.
- 15                   5. An apparatus according to claim 1, in which said second particle separation means is configured such that said air sample is divided into a plurality of branch streams.
- 20                   6. An apparatus according to claim 5, in which said second particle separation means is a plurality of high volume virtual impactors.
- 25                   7. An apparatus according to claim 6, in which said plurality of HVVIs are configured such that said fine particle part of said air sample separates into a plurality of divisions, each division comprised of particles within a specified particle size range, and such that each of said branch streams contains one of said particle component divisions.

-18-

8. An apparatus according to claim 5, in which said second particle separation means is configured such that flow volumes of said branch streams are substantially equivalent.

5 9. An apparatus according to claim 7, in which said HVVIs are configured such that said plurality of fine particle component divisions numbers four, and one of each said divisions comprises particles within the following ranges: 0-0.32  $\mu\text{m}$ , 0-0.8  
10  $\mu\text{m}$ , 0-1.5  $\mu\text{m}$ , and 0-2.5  $\mu\text{m}$ .

10. An apparatus according to claim 1, in which said intermediate filter means collects particles larger than 2.5  $\mu\text{m}$ .

11. An apparatus according to claim 1, in  
15 which said denuder means removes from said air sample at least two of the group consisting of said acid gas component, said basic gas components, and said volatile organic compounds.

12. An apparatus according to claim 1, in  
20 which said denuder means removes said acid gas component, said basic gas component, and said volatile organic compounds component.

13. An apparatus according to claim 1, in which said denuder means includes at least one of the  
25 group comprising: sodium carbonate film to remove said acid gas component; citric acid film to remove said basic gas component; and silicon grease to remove said volatile organic component.

14. An apparatus according to claim 1, in  
30 which said denuder includes at least two from the group comprising: sodium carbonate film to remove said acid

-19-

gas component; citric acid film to remove said basic gas component; and silicon grease to remove said volatile organic component.

15           15. An apparatus according to claim 1, in which said denuder includes sodium carbonate film to remove said acid gas component, citric acid film to remove said basic gas component, and silicon grease to remove said volatile organic component.

10           16. An apparatus according to claim 1, in which said denuder means includes a plurality of denuders through which a plurality of branch streams of said air sample flows.

15           17. An apparatus according to claim 16, in which at least one of said plurality of denuders includes at least one of the group comprising: sodium carbonate film to remove said acid gas component; citric acid film to remove said basic gas component; and silicon grease to remove said volatile organic  
20           component.

18. An apparatus according to claim 16, in which at least one of said plurality of denuders includes at least two from the group comprising: sodium carbonate film to remove said acid gas  
25           component; citric acid film to remove said basic gas component; and silicon grease to remove said volatile organic component.

19. An apparatus according to claim 16, in which at least one of said plurality of denuders  
30           includes sodium carbonate film to remove said acid gas component, citric acid film to remove said basic gas component, and silicon grease to remove said volatile organic component.

-20-

20. An apparatus according to claim 1, in which said fine filter means removes from said air sample particles as small as 0.001  $\mu\text{m}$ .

21. An apparatus according to claim 1, in which said fine filter means includes filter media comprised of at least one of the group consisting of Nucleopore, PTFE, PTFE-coated glass fiber, and quartz.

22. An apparatus according to claim 16, in which said fine filter means includes a plurality of filter sections.

23. An apparatus according to claim 22, in which filter media for said plurality of filter sections include at least one from the group consisting of Nucleopore, PTFE, PTFE-coated glass fiber, and quartz.

24. An apparatus according to claim 1, in which said SVOC collection means include polymer traps comprising at least at least one from the group consisting of XAD-2, PUFF, Tenax, and charcoal.

25. An apparatus according to claim 24, in which said means for collecting SVOCs includes a plurality of polymer traps.

26. An apparatus according to claim 25, in which said plurality of polymer traps includes at least one of the group consisting of XAD-2, PUFF, Tenax, and charcoal.

27. An apparatus according to claim 24, in which said polymer trap comprises a sandwich construction of a XAD-2 core and PUFF outer layers.

-21-

28. An apparatus according to claim 1, in which said flow control means includes at least one manually controlled butterfly valve.

29. An apparatus according to claim 1, in which said flow control means is electronically controlled.

30. An apparatus according to claim 1, in which said vacuum means is configured to draw said air sample through said housing at a flow volume of at least 35 cfm.

31. An apparatus used in separating and classifying the particle, acid gas, basic gas, VOC, and SVOC components of an air sample comprising:

- (a) a housing;
- 15 (b) denuder means that removes from said air sample at least one of the group consisting of said acid gasses, basic gasses, and volatile organic compounds, said denuder means being in a section of said housing;
- 20 (c) filter means that traps said particles contained in said air sample, said filter means being in a section of said housing downstream from said denuder means; and
- 25 (d) means for collecting SVOCs located in a section of said housing downstream from said filter means.

32. An apparatus according to claim 31, in which said denuder means includes a plurality of denuders.

33. An apparatus according to claim 31 in which said denuder means removes from the sample at least two of the group consisting of acid gasses, basic

-22-

gasses, and volatile organic compounds.

34. An apparatus according to claim 31, in which said denuder means removes from the sample acid gasses, basic gasses, and VOCs.

5           35. An apparatus according to claim 32, in which at least one of said plurality of denuders removes from the sample at least two of the group consisting of acid gasses, basic gasses, and VOCs.

10           36. An apparatus according to claim 32, in which at least one of said plurality of denuders removes from the sample acid gasses, basic gasses, and VOCs.

15           37. An apparatus according to claim 31, in which said filter means includes filter media including at least one from the group consisting of PTFE-coated glass fiber, Nucleopore, quartz, and PTFE.

38. An apparatus according to claim 31, in which said filter means includes a plurality of filters.

20           39. An apparatus according to claim 31, in which said plurality of filters includes filter media including at least one of the group consisting of PTFE-coated glass fiber, Nucleopore, quartz, and PTFE.

25           40. An apparatus according to claim 31, in which said SVOC collection means include a polymer trap comprising at least one of the group consisting of PUFF, XAD-2, Tenax, and charcoal.



-23-

41. An apparatus according to claim 31, in which said SVOC collection means includes a plurality of polymer traps.

42. An apparatus according to claim 31, in which said plurality of polymer traps comprises at least two of the group consisting of PUFF, XAD-2, Tenax, and charcoal.

43. An apparatus according to claim 40, in which at least one polymer trap includes a sandwich construction comprising a XAD-2 core and PUFF outer layers.

44. A method of separating and collecting particle, acid gas, basic gas, organic gas, and semi-volatile organic components in an air sample which comprises the steps of:

(a) drawing said air sample through a housing;

(b) separating said air sample into a first large particle part and a second part;

(c) separating said second part of said air sample into a coarse part and a fine part;

(d) removing from said air sample at least one of the group comprising acid gasses, basic gasses, and organic gasses;

(e) trapping said fine part of said particle component in a fine filter means; and

(f) removing semi-volatile organic compounds.

45. A method according to claim 44, in which said coarse part of said particle component is trapped in a coarse filter means;

-24-

46. A method according to claim 44, in which said air sample is drawn through said housing at a volumetric flow rate of at least 35 cfm.

47. A method according to claim 44, in which  
5 the flow rate of said air sample drawn through said housing is monitored by flow rate control means.

48. A method according to claim 47, in which said flow rate control means is manually monitored.

49. A method according to claim 47, in which  
10 said flow rate control means is monitored electronically.

50. A method according to claim 44, in which said large particle part of said particle component of said air sample is comprised of particles larger than  
15 10  $\mu\text{m}$ .

51. A method according to claim 44, in which said large particle part is separated utilizing a two-step inlet.

52. A method according to claim 44, in which  
20 said separation of said particle component into a coarse part and a fine part utilizes a high volume virtual impactor.

53. A method according to claim 44, in which said coarse part of said particle component comprises  
25 particles larger than 2.5  $\mu\text{m}$ .

54. A method according to claim 44, in which said air sample is divided into branch streams.

-25-

55. A method according to claim 54, in which the flow volumes of said branch streams are substantially equivalent.

56. A method according to claim 54, in which  
5 said division of said air sample into branch streams occurs during said separation of said particle component into said coarse part and said fine part.

57. A method according to claim 54, in which  
10 said division of said air sample into branch streams occurs just prior to said removal of at least one of the group consisting of acid gasses, basic gasses, and volatile organic compounds.

58. A method according to claim 56, in which  
15 said separation of said air sample into said intermediate part and said fine part utilizes a plurality of HVVIs.

59. A method according to claim 58, in which  
20 said fine part is separated into a plurality of divisions based on particle size.

60. A method according claim 59, in which  
said divisions of said fine part have separation cutpoints of 0.32 $\mu$ m, 0.8 $\mu$ m, 1.5 $\mu$ m, and 2.5 $\mu$ m.

61. A method according to claim 44, in which  
25 two of the group comprising acid gasses, basic gasses, and volatile organic compounds are removed.

62. A method according to claim 44, in which  
acid gasses, basic gasses, and VOCs are removed.

-26-

63. A method according to claim 44, in which at least one of the group consisting of sodium carbonate, citric acid, and silicon grease is utilized.

64. A method according to claim 44, in which  
5 at least two of the group consisting of sodium carbonate, citric acid, and silicon grease are utilized.

65. A method according to claim 44, in which sodium carbonate, citric acid, and silicon grease are  
10 utilized.

66. A method according to claim 44, in which a plurality of denuders is utilized.

67. A method according to claim 44, in which at least one of said plurality of denuders removes from  
15 the sample at least one of the group consisting of acid gasses, basic gasses, and volatile organic compounds.

68. A method according to claim 44, in which at least one of said plurality of denuders removes from the sample at least two of the group consisting of acid  
20 gasses, basic gasses, and volatile organic compounds.

69. A method according to claim 44, in which at least one of said plurality of denuders removes from the sample acid gasses, basic gasses, and volatile organic compounds.

25 70. A method according to claim 44, in which said filter means utilizes a filter media, said filter media comprised of at least one of the group consisting of PTFE-coated glass fiber, Nucleopore, quartz, and PTFE.

-27-

71. A method according to claim 70, in which a plurality of filter media sections is utilized.

72. A method according to claim 71, in which said plurality of filter media sections includes at least one of the group consisting of PTFE-coated glass fiber, Nucleopore, quartz, and PTFE.

73. A method according to claim 44, in which said SVOCs are removed by at least one of the group consisting of PUFF, XAD-2, Tenax, and charcoal.

10 74. A method according to claim 73, in which said SVOCs are removed by at least two of the group consisting of PUFF, XAD-2, Tenax, and charcoal.

75. A method according to claim 44, in which a plurality of SVOC collection means are utilized.

15 76. A method according to claim 74, in which at least one of said plurality of SVOC collection means includes at least one of the group consisting of PUFF, XAD-2, Tenax, and charcoal.

20 77. A method of measuring the VOC concentration in an air sample comprising the steps of:

(a) dividing said air sample into a plurality of substantially equivalent branch flows;

(b) passing at least one of said branch flows through denuder means that remove volatile organic compounds;

(c) passing said plurality of branch flows through a similar plurality of filter means that traps the particles present in said sample;

30 (d) passing said plurality of branch flows through a similar plurality of SVOC collection means; and

-28-

(e) removing and analyzing the composition and concentration of the filtrate trapped by said filter means filtrate and the SVOC components trapped by said SVOC collection means for each branch flow.

- 5           78. A method according to claim 77, in which said plurality of branch flows equals two.

1/3

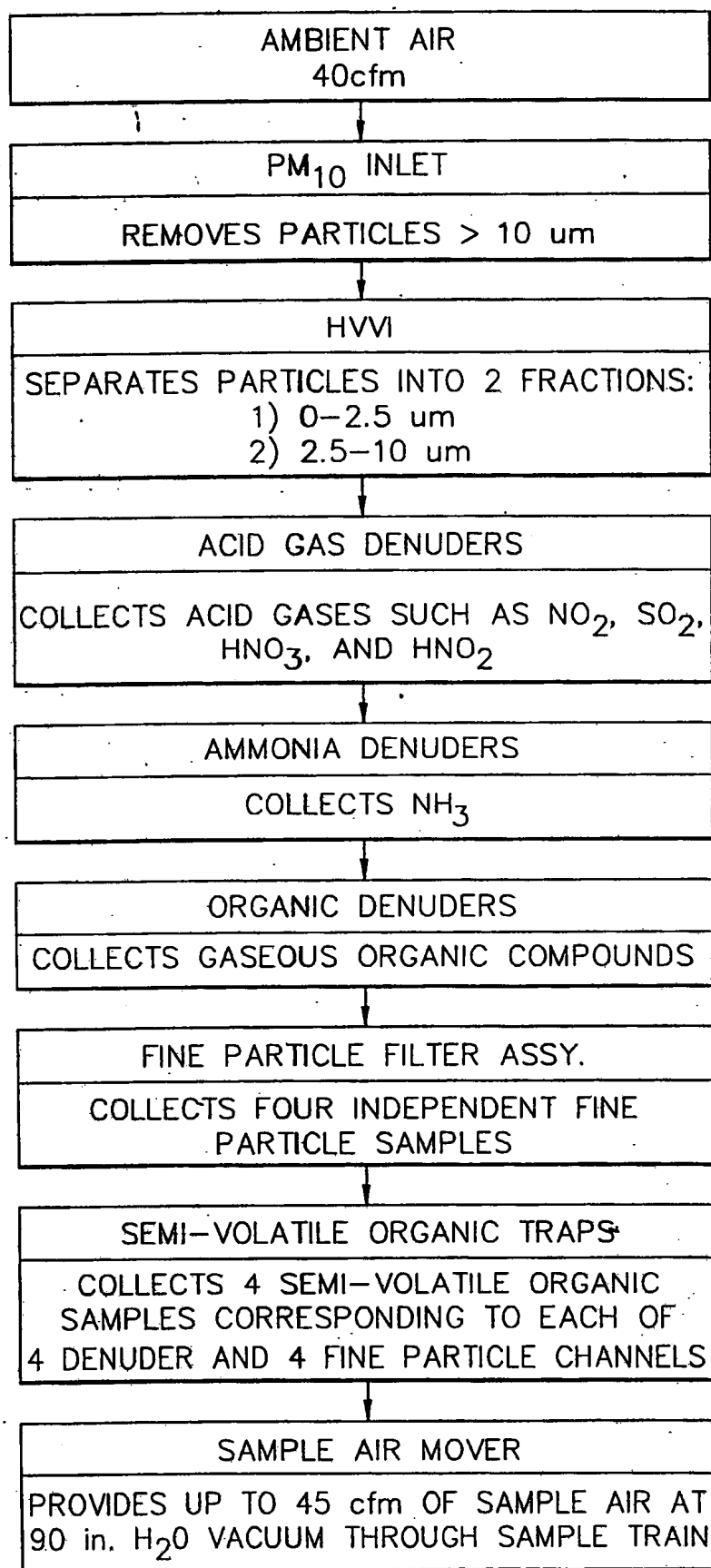


FIG. 1.

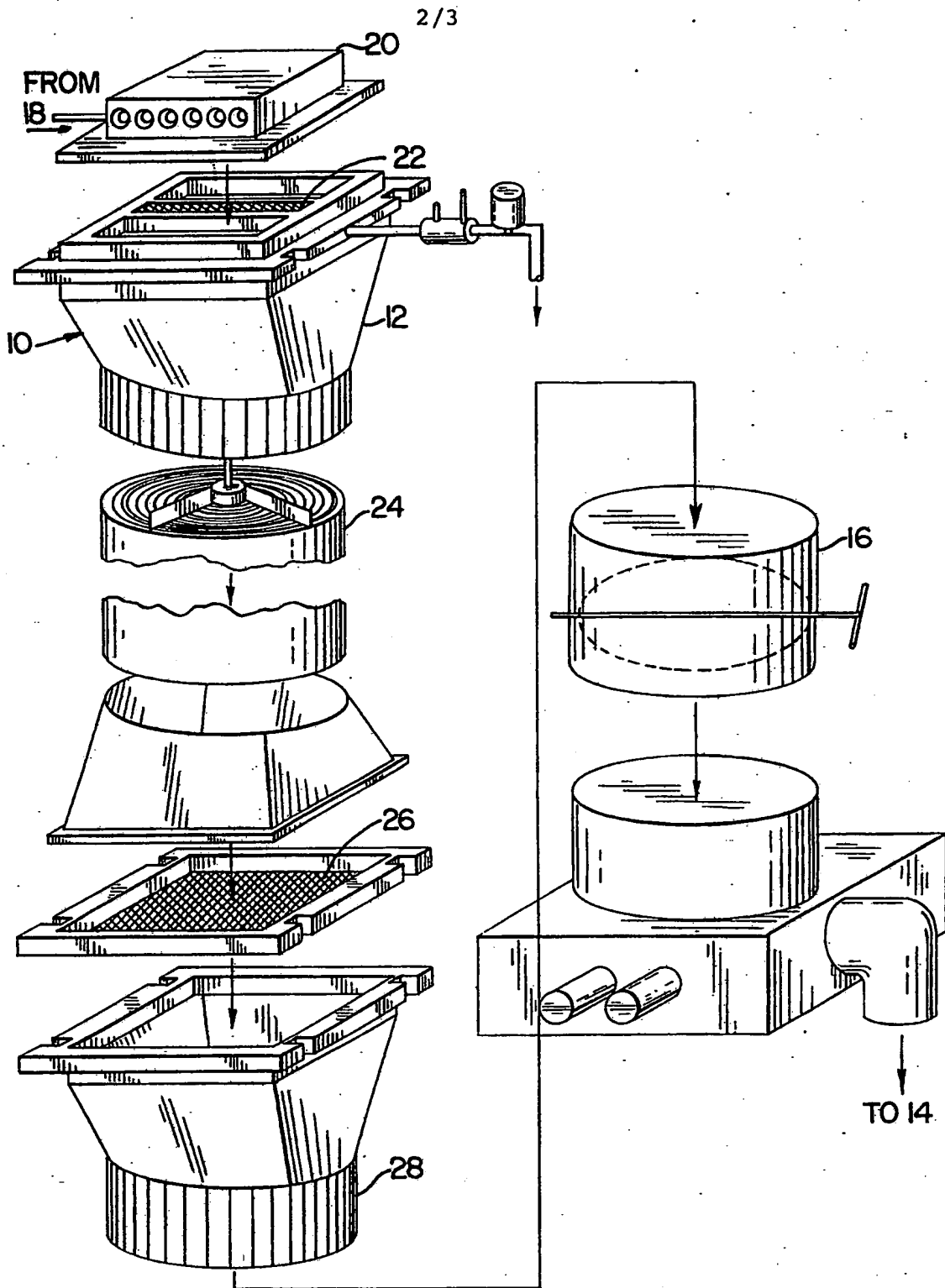


FIG. 2.

SUBSTITUTE SHEET



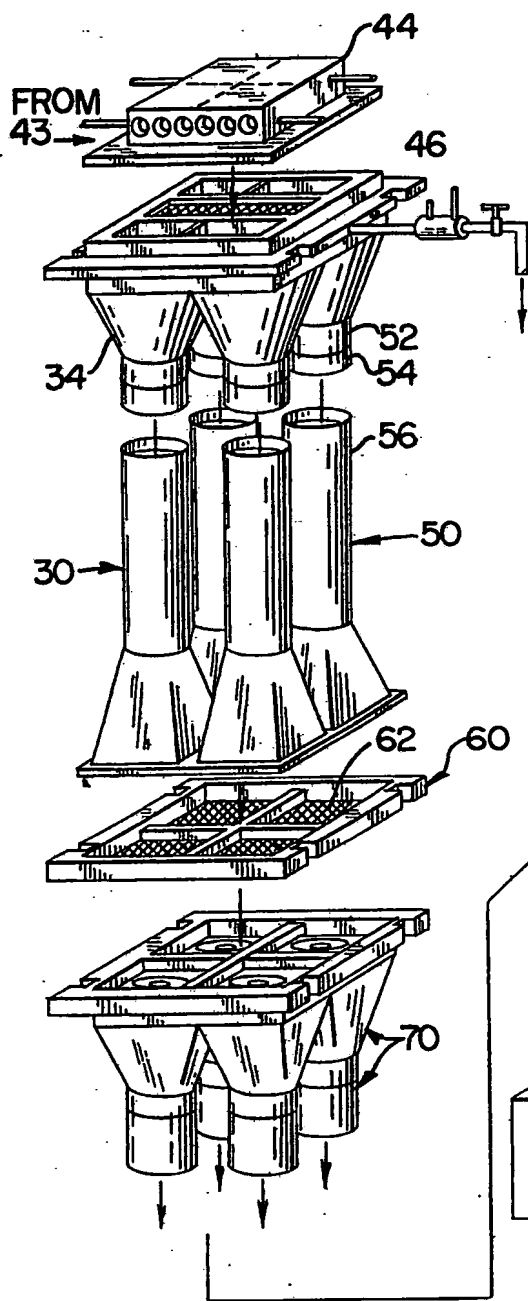


FIG. 3.

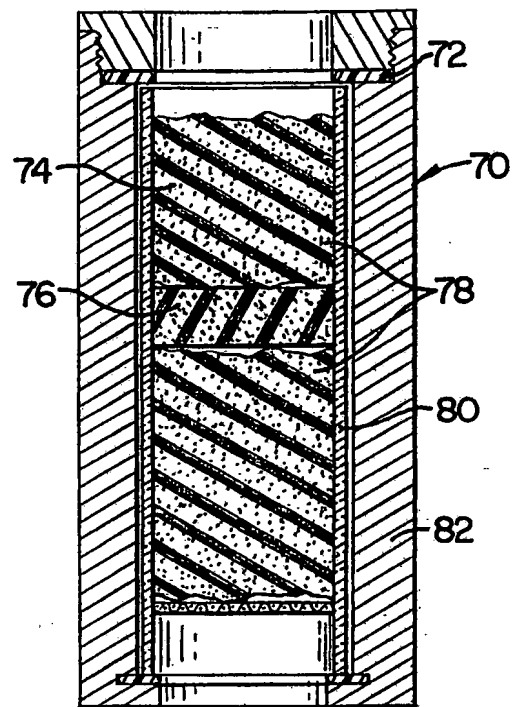
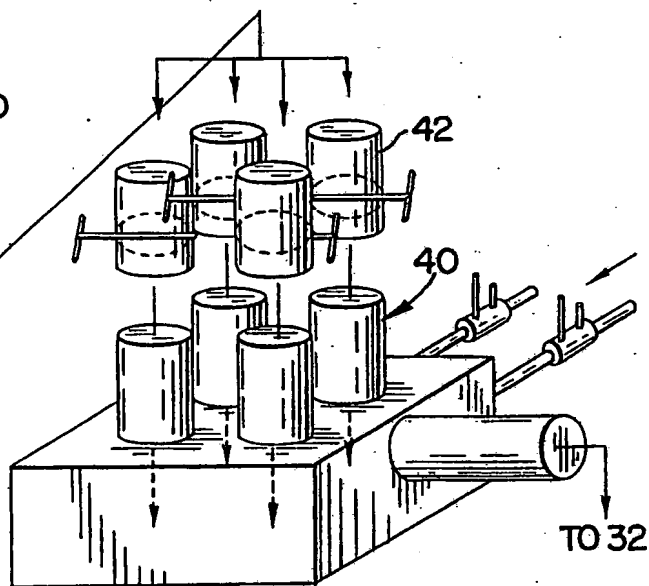


FIG. 4.



## INTERNATIONAL SEARCH REPORT

PCT/US92/08532

**A. CLASSIFICATION OF SUBJECT MATTER**

IPC(5) :B01D 35/157; B01D 51/10

US CL :055/001; 055/270

According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 055/018,097; 73/031.07

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	V.A. MARPLE ET AL., "High-volume Impactor for Sampling Fine and Coarse Particles: <u>I. Air Waste Manage. Assoc.</u> 40: 762-767 (1990). See Figure 3 and its associated description.	1-78
Y	R.W. COUTANT ET AL., "Design and Performance of a High-Volume Compound Annular Denuder", <u>Atmospheric Environment</u> , Vol. 23, No. 10, 2205-2211, 1989, Fig. 3 and its associated description.	31-36,38, 44-46,50- 70,77-78
Y	US, A, 4,432,248 (LALIN) 21 February 1984, See dampers 95 and 97.	1-20,29-30, 39-43,47-49



Further documents are listed in the continuation of Box C.



See patent family annex.

* Special categories of cited documents:	"T"	later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"A" document defining the general state of the art which is not considered to be part of particular relevance	"X"	document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"E" earlier document published on or after the international filing date	"Y"	document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	"Z"	document member of the same patent family
"O" document referring to an oral disclosure, use, exhibition or other means		
"P" document published prior to the international filing date but later than the priority date claimed		

Date of the actual completion of the international search

08 DECEMBER 1992

Date of mailing of the international search report

04 JAN 1993

Name and mailing address of the ISA/  
Commissioner of Patents and Trademarks  
Box PCT  
Washington, D.C. 20231

Authorized officer

ROBERT H. SPITZER

Facsimile No. NOT APPLICABLE

Telephone No. (703) 308-3794

## INTERNATIONAL SEARCH REPORT

International application No.

PCT/US92/08532

## C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	R.G. LEWIS ET AL., "Modification and Evaluation of a High-Volume Air Sampler for Pesticides and Semivolatile Industrial Organic Chemicals", <u>Anal. Chem.</u> March, 1982, pp. 592-594. See Figure 2.	21-28, 71-76
A	R.W. COUTANT ET AL., "Phase Distribution and Artifact Formation in Ambient air sampling for Polynuclear Aromatic Hydrocarbons", <u>Atmospheric Environment</u> , Vol. 22, No. 2, pp. 403-409, 1988.	1-78
A	US, A, 3,252,323 (TORGESON) 24 May 1966.	1,44,77
A	US, A, 4,902,318 (STEVENS ET AL.) 20 February 1990.	25-26,40-41, 43
A	US, A, 5,006,227 (BEHM ET AL.) 09 April 1991.	1-78
A	US, A, 4,670,135 (MARPLE ET AL.) 02 June 1987.	1-78
A	US, A, 4,569,235 (CONKLE ET AL.) 11 February 1986.	1-78
A	US, A, 4,961,916 (LESAGE ET AL.) 09 October 1990.	1-78
A	US, A, 4,274,846 (SMITH) 23 June 1981.	1-78
A	US, A, 3,983,743 (OLIN ET AL.) 05 October 1976.	1-78

# INTERNATIONAL SEARCH REPORT

International application No.  
PCT/US92/08532

## Box I Observations where certain claims were found unsearchable (Continuation of item 1 of first sheet)

This international report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:  
because they relate to subject matter not required to be searched by this Authority, namely:
2. ☐ Claims Nos.:  
because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:
3. ☐ Claims Nos.:  
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

## Box II Observations where unity of invention is lacking (Continuation of item 2 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:  
Please See Extra Sheet.

1. ☐ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims.
2. ☒ As all searchable claims could be searched without effort justifying an additional fee, this Authority did not invite payment of any additional fee.
3. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:
4. ☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

Remark on Protest

- ☐ The additional search fees were accompanied by the applicant's protest.  
☐ No protest accompanied the payment of additional search fees.

# INTERNATIONAL SEARCH REPORT

International application No.  
PCT/US92/08532

## BOX II. OBSERVATIONS WHERE UNITY OF INVENTION WAS LACKING

This ISA found multiple inventions as follows:

Group I. Claims 1-43, drawn to apparatus for separating and collecting components from an air sample, classified in class 55, subclass 270.

Group II. Claims 44-76, drawn to a method for separating and collecting components of an air sample, classified in class 55, subclass 1.

Group III. Claims 77-78, drawn to a method of analyzing the makeup of an air sample, classified in class 73, subclass 31.07.

The inventions of Groups II and I are related as process and apparatus for its practice.

The apparatus of Group I, as claimed, can be used to practice another and materially different process, such as separation of gas into components for purpose other than analysis.

The inventions of Groups II and III are related as combination and subcombination. The combination, as claimed, does not require the particulars of the subcombination as claimed because the process of Group III does not require the same to be separated into a coarse part and a fine part or the removal of the acid and basic gases. The subcombination (Group III) has separate utility such as separation of an air sample into its components for reasons other than analysis, which would include separation for purification purposes.